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L8: Entry 6 of 7

File: DWPI

Nov 29, 1977

DERWENT-ACC-NO: 1978-05139A
DERWENT-WEEK: 197803
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TITLE: Stabilised aq. suspensions of binapacryl - for mildew and mite control, contg. alizarin and a water-soluble aluminium cpd., having a pH of 5 to 9

PATENT-ASSIGNEE:

ASSIGNEE

CODE

SANKYO CO LTD

SANY

PRIORITY-DATA: 1976JP-0060747 (May 26, 1976)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

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MAIN-IPC

JP 52143224 A

November 29, 1977

000

INT-CL (IPC): A01N 9/20; A01N 17/00

ABSTRACTED-PUB-NO: JP 52143224A

BASIC-ABSTRACT:

Stabilised aq. suspensions of Binapacryl (I) contg. alizarin (II) and a water-soluble Al cpd. (III) have a pH of 5.0-9.0. If desired, a water-soluble polymer cpd. is added to the aq. suspension. When the pH exceeds 7, Binapacryl becomes liable to be decomposed. The suspension contains 10-50 wt. %, pref. 20-40 wt. % (I). (II) is contained in an amt. of 0.01-3 wt. %, pref. 0.05-1 wt. %. (III) is suitably aluminium chloride, basic aluminium acetate (pref.), aluminium sulphate or alum. (III) is present in an amt. of 0.05-5 wt. %, pref. 0.1-2 wt. %. Blend ratio of (III) to (II) is pref. 1:1-2.

Binapacryl is used to control powdery mildew and mites. Aq. suspensions of (I) have the defect that a ppte. layer, difficult to suspend again, is formed during a storage and that this deterioration is promoted above 40 degrees C. This novel suspension is stable under high temp. conditions for long periods.

TITLE-TERMS: STABILISED AQUEOUS SUSPENSION MILDEW MITE CONTROL CONTAIN ALIZARIN WATER SOLUBLE ALUMINIUM COMPOUND PH

DERWENT-CLASS: A97 C03

CPI-CODES: A08-M02; A12-W04; A12-W12; C05-A01B; C08-D02; C10-G02; C12-A02; C12-B04; C12-M03; C12-M06;

CHEMICAL-CODES:

Chemical Indexing M2 *01*

Fragmentation Code

J2 H3 H7 M210 M213 M214 M232 M240 M260 M281
M313 M314 M320 G100 M531 J241 J242 H341 H342 H343
H721 M431 P002 P241 P242 M510 P330 P331 P332 M520
M540 M782 R003 R023 R024 M414 M902

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H721 M431 P002 P241 P242 M510 P330 P331 P332 M520
M540 M782 R003 R023 R024 M414 M902

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審査請求 未請求

(全 4 頁)

⑮安定化されたビナパクリルの水性懸濁剤

⑯発明者 藤本昌彦

滋賀県野洲郡野洲町大字野洲10

41 三共株式会社農業研究所内

⑰特 願 昭51—60747

⑱出 願 昭51(1976)5月26日

⑲出 願 人 三共株式会社

⑳発明者 中村利家

東京都中央区日本橋本町3丁目

滋賀県野洲郡野洲町大字野洲10

1番地の6

41 三共株式会社農業研究所内

㉑代理人 弁理士 樫出庄治

PTO 2003-2986

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明 細 書

1 発明の名称

安定化されたビナパクリルの水性懸濁剤

2 特許請求の範囲

1. アリザリンと水溶性アルミニウム化合物を含有し且つ懸濁剤のpHが5.0～9.0であることを特徴とする安定化されたビナパクリルの水性懸濁剤。

3 発明の詳細な説明

本発明は、安定化されたビナパクリルの水性懸濁剤にかかわり、さらに詳しくはアリザリンと水溶性アルミニウム化合物を含有し、且つ懸濁剤のpHが5.0～9.0であることを特徴とする安定化されたビナパクリルの水性懸濁剤に関するものである。

ビナパクリル(化学名: 2,4-ジニトロ-6-セカンダリーブチルフェニルジメチルアクリレート)は、農園栽培作物に寄生し、害作用を及ぼすうどん粉病の防除に、またダニ類に対しても有効な化合物である。

本化合物は水に難溶性であり、水を分散媒とする水性懸濁剤を調製することができるが、貯蔵中に懸濁剤中の粒子が結晶状に粒子成長して沈降し、容器底部に強固な沈殿層(ヘッドゲイキング)を形成したり、分散粒子同士が互いに強く凝集し懸濁剤を水等の散布用希釈液に加えた際に、個々の粒子に再分散し難い凝集塊(コアグュレーション)を形成する等、物理性の劣化する現象がみられる。特にこれらの傾向は40℃以上の高温時に著しく促進され、懸濁剤として致命的な欠陥となるためビナパクリルの水性懸濁剤を実用に供する上で重要な問題であつた。

農業懸濁剤の貯蔵時における物理性の劣化防止に関しては、これまで種々の方法が試みられてきた。例えば、水性懸濁剤に関しては、懸濁媒体として硫酸アンモニウム、磷酸二水素ナトリウム等の無機塩の水性濃厚液を用いて懸濁液の安定化を図る方法(特開昭50-76236号)が知られている。しかしながらこの方法では、ビナパクリル水性懸濁剤の貯蔵時における安定

化効果は満足できるものではなかつた。また、アゾ染料-錯化合物及びフタロシアニン染料-錯化合物系から成る金属錯化合物染料を水性分散物に添加した2-アルキル-4,6-ジニトロフェノールエステル類の安定化法(特開昭49-126635号)は、必ずしもすべてに効果が的確であるとは云えず、このため一層確実な安定化手段の出現が強く望まれていた。更に、特開昭50-63141号にはカルボン酸アミド類を水不溶性の有機溶媒中に分散せしめた懸濁剤の場合に、アリザリンとアルミニウム化合物を添加した油性懸濁剤の貯蔵安定化法が記載されているが、水性懸濁剤については何等開示していない。

一般に分散媒として水を使用した水性懸濁剤の方が油性懸濁剤より植物に対する薬害が少なく、製造時の安全性、取扱いの便利さ等から農薬製剤としてはより有利である。

本発明者らは、ビナバクリルを水を分散媒とする水性懸濁剤に調製したときの貯蔵時におけ

(3)

であると云える。従つて水性懸濁剤の貯蔵時における安定性の目安は、40~50℃の高温条件下で比較的短期間(2~4週間)観察することにより知ることができる。

本発明の要旨は、ビナバクリルの水を分散媒とした水性懸濁剤中にアリザリンと水溶性アルミニウム化合物を添加し、且つpHを5~9の酸性とし、更に要すれば水溶性高分子化合物を添加することにある。即ち、懸濁剤のpHが4以上になるとアリザリンのアルミニウムキレート化合物の形成が促進され、pHが5以上になるとアリザリンのアルミニウムキレート化合物が分散粒子表面に効率よく吸着されることにより、分散粒子の高温時(40~50℃)における粒子成長が抑制され、また、粒子のコアギネレーションも抑制されること、更に、水溶性高分子化合物を少量添加することにより懸濁液全体が弱い凝集性“フロツキネーション”

(Flocculation)を帯びることにより、貯蔵中における安定性が著しく良くなると考えられる。

(5)

る物理性の劣化を防止すべく鋭意研究を重ねた結果、水性懸濁剤中にアリザリンおよび水溶性のアルミニウム化合物を添加し、更にpHを5~9とし、更に要すれば水溶性高分子化合物を少量添加することにより、貯蔵中の沈降による凝固で再懸濁が困難な沈殿層の生成等、物理性の劣化がことごとく顕著に改善され、安定な水性懸濁剤が得られることを見出した。

この場合にアリザリンと構造類似のアリザリンレッド8(化学名:3,4-ジヒドロキシアントラキノン-2-スルホン酸ナトリウム)の添加では全く安定化効果は認めることができず、本発明のビナバクリルとある特定の化合物の組合せが特異的な効果を示したことは全く意外な事実である。

ビナバクリルの水性懸濁剤の貯蔵時における物理性の劣化は、30℃以下では比較的發展であるが、40℃以上では著しく促進される。一般的に、40℃以上の高温条件下で物理性の劣化の少ないもの程、30℃以下においても僅少

(4)

ビナバクリルの水性懸濁剤の場合は、系全体が分散粒子同士緩やかに凝集をなし、軽く振盪または攪拌することによつて個々の粒子に再分散する状態にあることが、本発明の目的を達成する上で極めて重要なことである。このためには懸濁剤のpHが5以上、好適にはpH6~9の中性乃至弱アルカリ性であることが必須である。一方、懸濁剤のpHが9を越えるとビナバクリルが分解され易くなり、好ましくない。

懸濁剤はビナバクリルを10~50重量%含有し、好ましくは20~40重量%含有する。

アリザリンは懸濁剤の0.01~3重量%、好適には0.05~1重量%含有する。

水溶性アルミニウム化合物は、塩化アルミニウム、塩基性酢酸アルミニウム、硫酸アルミニウム、ミヨウバン等から選択される。好適な水溶性アルミニウム化合物は塩基性酢酸アルミニウムであり、懸濁剤の0.05~5重量%、好適には0.1~2重量%含有する。懸濁剤中のアリザリンに対する水溶性アルミニウム化合物の配合

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比(重量)は1対1~2が好ましい。

アリザリンと水溶性アルミニウム化合物は粉碎の前に添加することが好ましいが、粉碎後ゾル剤調製時に添加してもよい。ただしアリザリンは水溶性が低いので、粉碎後に添加するときはあらかじめ微粉碎するか、あるいは水酸化アルカリ水溶液に溶解させて添加する。

水溶性高分子化合物は、メチルセルロース、ヒドロキシエチルセルロース、ポリビニルアルコール、可溶性デンプン等から選択される。好適な水溶性高分子化合物はヒドロキシエチルセルロースであり、懸濁剤の0.05~5重量%、好適には0.1~2重量%含有する。

また、本発明の水性懸濁剤の調製に際し、混濁、分散等の目的でポリオキシエチレンアルキルエーテル、ポリオキシエチレンアルキルアリールエーテル等の非イオン性界面活性剤やラウリルベンゼンスルホン酸ナトリウム、ジアルキルスルホコハク酸ナトリウム、リグニンスルホン酸塩、ナフタリン-ホルムアルデヒド縮合物

(7)

アリザリン	0.3 部
塩基性酢酸アルミニウム	0.3 部
水酸化ナトリウム (pH調整用)	0.3 部
エチレングリコール	6.0 部
ヒドロキシエチルセルロース	0.5 部
水	53.6 部

ビナバクリル、リグニンスルホン酸カルシウム、アリザリン、塩基性酢酸アルミニウム、水酸化ナトリウム、エチレングリコールおよび水4.0部を混合して懸濁液とし、アトライター(三井・三池製作所製、MA-18型)の粉碎槽に入れ、粉碎メディアとして直径4mmのステンレス製ボールを加えて2時間粉碎した。得られた懸濁液にヒドロキシエチルセルロースを残余の水に溶解した溶液を加えて再度混合し、ビナバクリルの水性懸濁剤を得た。このもののpHは7.5であつた。

参考例1

実施例1の処方よりアリザリン0.3部および塩基性酢酸アルミニウム0.3部を除き、代わり

(9)

のスルホン酸塩等の陰イオン性界面活性剤が懸濁剤の0.01~10重量%の範囲で用いられる。

更に、エチレングリコール、ジエチレングリコールや硫酸アンモニウム、塩化アンモニウム、硫酸ナトリウム等の無機塩のいずれかを凍結防止剤として懸濁剤中に0~10重量%含有させることもできる。

本発明の安定化されたビナバクリルの水性懸濁剤の製造はきわめて簡単であり、次の一般的な方法のいずれかを用いて製造される。(1)ビナバクリルをあらかじめ乾式粉碎機で微粉碎した後、水性分散媒中に懸濁させる。(2)ビナバクリルを水性分散媒中に懸濁させた後、湿式粉碎機で微粉碎化する。

以下に実施例、参考例および試験例を挙げて本発明を更に説明する。(文中、単に部とあるのは全て重量部をあらわす。)

実施例1

ビナバクリル	53.0 部
リグニンスルホン酸カルシウム	6.0 部

(8)

に同一重量の水を添加する以外は、実施例1と全く同様にしてビナバクリルの水性懸濁剤を得た。このもののpHは7.6であつた。

参考例2

実施例1の処方より水酸化ナトリウム0.3部を除き、代わりに同一重量の水を添加する以外は、実施例1と全く同様にしてビナバクリルの水性懸濁剤を得た。このもののpHは3.7であつた。

参考例3

実施例1の処方より水酸化ナトリウムを0.2部減じ、代わりに同一重量の水を添加する以外は、実施例1と全く同様にしてビナバクリルの水性懸濁剤を得た。このもののpHは5.0であつた。

参考例4

実施例1の処方よりヒドロキシエチルセルロースを除き、代わりに同一重量の水を添加する以外は、実施例1と全く同様にしてビナバクリルの水性懸濁剤を得た。このもののpHは7.5

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であつた。

試験例 1

本発明による実施例 1 の水性懸濁剤および参考例 1～4 で得た水性懸濁剤を、内径 4.5 cm、高さ 8 cm のシリンダーに高さ 7 cm の位置まで加え、ポリエチレンの塗をして 50℃ の恒温室に 2 週間静置した。水性懸濁剤中の軟凝集粒子層の高さ (cm) を 7 で除した数値を沈降容積比とし、更にシリンダーを手で振り混ぜて、軟凝集粒子層が再び流動性を示す性質を再流動分散性とした。尚、上記軟凝集粒子層の上の層は透明な水性分散質層となつている。再流動分散性の評価は次の基準により表示した。

[O] ; シリンダーを軽く振つただけで直ちに水性懸濁剤が流動性を示す。

[Δ] ; シリンダーを強く振り混ぜなければ流動性を示さない。

[x] ; シリンダーの底部に強固なヘッドケーキングを形成しており、シリンダーを強く振り混ぜても流動性を示さない。

	沈 降 容 積 比		再 流 動 分 散 性	
	経時初期	加熱試験 (50℃, 14日間)	経時初期	加熱試験 (50℃, 14日間)
実施例 1	1.00	0.92	○	○
参考例 1	1.00	0.55	○	x
" 2	1.00	0.56	○	x
" 3	1.00	0.75	○	Δ
" 4	1.00	0.70	○	Δ

特許出願人 三共株式会社
代理人 弁理士 檀 出 庄 治

(11)

(12)

PTO 03-2986

Japanese Patent

Document No. Sho 52-143224

AQUEOUS SUSPENSION OF STABILIZED BINAPACRYL

[Anteikasareta Binapakuriru No Suisei Kentakuzai]

Toshiie Nakamura and Masahiko Fujmoto

UNITED STATES PATENT AND TRADEMARK OFFICE

Washington, D.C.

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Fujimoto

Applicant : Sankyo Co., Ltd.

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Foreign Language Title : Anteikasareta Binapakuriru No
Suissei Kentakuzai

English Title : AQUEOUS SUSPENSION OF STABILIZED
BINAPACRYL

Specification

1. Title of the invention

Aqueous Suspension of Stabilized binapacryl

2. Claim

1. An aqueous suspension of a stabilized binapacryl, characterized by the fact that an alizarine and a water-soluble aluminum compound are included; and pH of a suspension is 5.0-9.0.

3. Detailed explanation of the invention

The present invention pertains to an aqueous suspension of a stabilized binapacryl. More specifically, the present invention pertains to an aqueous suspension of a stabilized binapacryl characterized by an alizarine and a water-soluble aluminum compound are included and pH of a suspension is 5.0-9.0.

¹ Numbers in the margin indicate pagination in the foreign text.

Binapacryl (chemical name: 2,4-dinitro-6-sec-butylphenyldimethyl acrylate) is a compound that is parasitic to agricultural and horticultural corps and also effective for controlling harmful actions and Boston powder disease and Acarina.

This compound is slightly soluble in water, and an aqueous suspension in which water is a dispersion medium can be prepared. However, the particles in the suspension grow in a crystalline shape during its storage and precipitates, so that a strong precipitate layer (hard caking) is formed at the bottom of a container. Also, the dispersed particles are strongly adhered to each other, and when the suspension is added to a diluent for spraying water, etc., aggregates (coagulation) which are difficult to be re-dispersed in each particle are formed, so that the physical properties are degraded. In particular, since these tendencies were considerably accelerated at high temperature of 40°C or higher and became critical defects as a suspension, they were important issues in supplying the aqueous suspension of the binapacryl to practical uses.

On the degradation prevention of the physical properties of the agricultural chemical suspension during the storage, various methods have been attempted up to now. For example, as an aqueous suspension, a method for stabilizing a suspension by

using an aqueous rich solution of inorganic salts such as ammonium sulfate and sodium dihydrogen phosphate as a suspension medium (Japanese Kokai Patent Application No. Sho 55[1980]-76236) is known. However, in the method, the stabilization effect could not be met during the storage of the aqueous /2 binapacyl suspension. Also, in a method for stabilizing 2-alkyl-4,6-dinitrophenol esters in which a metal complex compound dye composed of azo dye-complex compound and phthalocyanine dye-complex compound systems (Japanese Kokai Patent Application No. Sho 49[1974]-126635), it cannot be said that all the effects are not necessarily accurate, and for this reason, the advent of a more accurate stable means has been in demand. Furthermore, in Japanese Kokai Patent application No. Sho 50[1975]-63141, a storage stabilization method of an oily suspension in which alizarine and an aluminum compound are added to a suspension in which amide carboxylates are dispersed into a water-soluble organic solvent is described. However, no aqueous suspension is mentioned at all.

In general, the harmful effects of an aqueous suspension, which uses water as a dispersion medium, rather than an oily suspension on plants are less, and the aqueous suspension is more favorable as an agricultural chemical in terms of manufacture safety, handling convenience, etc.

In these inventors, when the binapacryl was prepared as an aqueous suspension using water as a dispersion medium, lots of earnest researches were repeated to prevent the degradation of the physical properties during the storage of the aqueous suspension. As a result, it was discovered that if an alizarine and a water-soluble aluminum compound were added to an aqueous suspension, pH was adjusted to 5-9, and a small amount of water-soluble high-molecular compound was added, the degradation of the physical properties such as generation of a precipitated layer, which was difficult to be re-suspend, by the sedimentation during the storage was respectively markedly improved, so that a stable aqueous suspension could be obtained.

In this case, in the addition of an alizarine red S (chemical name: sodium 3,4-dihydroxyanthraquinone-2-sulfonate) with a structure similar to that of the alizarine, a stabilization effect cannot be recognized at all, and it is unexpected that the combination of the binapacryl of the present invention and a specific compound exhibits a specific effect.

The degradation of the physical properties during the storage of the aqueous suspension of the binapacryl is relatively mild at 30°C or lower, however it is considerably accelerated at 40°C or higher. In general, it can be said that if the degradation of the physical properties under a high

temperature condition of 40°C or higher is small, the degradation is also slight at 30°C or lower. Therefore, the estimation of the stability during the storage of the aqueous suspension can be detected by observing for a relatively short period (2-4 weeks) under a high temperature condition of 40-50°C.

The essence of the present invention is that an alizarine and a water-soluble aluminum compound are added into an aqueous suspension using water of binapacryl as a dispersion medium, pH is adjusted to 5-9, and if necessary, a water-soluble high-molecular compound is added. In other words, if pH of the suspension is 4 or more, the formation of an aluminum chelating compound of the alizarine is accelerated, and the if pH is 5 or more, the aluminum chelating compound of the alizarine is adsorbed with good efficiency to the surface of the dispersed particles, so that the particle growth at a high temperature (40-50°C) of the dispersed particles is suppressed and the coagulation of the particles is also suppressed. Furthermore, with a small amount of water-soluble high-molecular compound, the entire suspension exhibits a weak cohesive "flocculation." Thus, it is considered that the stability during the storage is markedly improved.

In the aqueous suspension of the binapacryl, the dispersed particles are mildly flocculated with each other in the entire

system, so that the binapacryl is re-dispersed into each particle by lightly shaking or stirring. This is very important to achieve the purpose of the present invention. For this reason, it is essential that pH of the suspension is 5 or more, preferably pH is 6-9, that is, neutral to weak alkaline. On the other hand, pH of the suspension exceeds 9, the binapacryl is easily decomposed, which is not preferable.

The suspension includes 10-50 wt% binapacryl, preferably 20-40 wt%.

The alizarine is included at 0.01-3 wt%, appropriately 0.05-1 wt% of the suspension.

The water-soluble aluminum compound is selected from aluminum chloride, basic aluminum acetate, aluminum sulfate, alum, etc. The water-soluble aluminum compound is appropriately a basic aluminum acetate and is included at 0.05-5 wt%, appropriately 0.1-2 wt% of the suspension. The mixture ratio (weight) of the water-soluble aluminum compound to the alizarine in the suspension is preferably 1:1-2. /3

The alizarine and the water-soluble aluminum compound are preferably added before crushing, however they may also be added during the formation of a sol agent after crushing. However, since the alizarine has a low water solubility, when it is added

after crushing, it is finely crushed in advance or dissolved in an aqueous alkali hydroxide solution and added.

The water-soluble high-molecular compound is selected from methyl cellulose, hydroxyethyl cellulose, polyvinyl alcohol, and soluble starch, etc. The water-soluble high-molecular compound is appropriately a hydroxyethyl cellulose and is included at 0.05-5 wt%, appropriately 0.1-2 wt% of the suspension.

Also, in preparing the aqueous suspension of the present invention, a nonionic surfactant such as polyoxyethylene alkyl ether and polyoxyethylene alkylallyl ether and an anionic surfactant such as sodium laurylbenzenesulfonate, sodium dialkylsulfosuccinate, lignin sulfonate, and sulfonate of naphthalin-formaldehyde condensate are used in a range of 0.01-10 wt% of the suspension.

Furthermore, any of inorganic salts such as ethylene glycol, diethylene glycol, ammonium sulfate, ammonium chloride, and sodium sulfate can be included at 0-10 wt% as an antifreezing agent in the suspension.

The manufacture of the aqueous suspension of the stabilized binapacryl of the present invention is very simple, and any of the following general methods is used in the manufacture. (1) A method that finely crushes the binapacryl in advance by a dry crusher and suspends it into an aqueous dispersion medium. (2)

A method that suspends the binapacryl into an aqueous dispersion medium and finely crushes it by a wet crusher.

Next, referring to an application example, referential examples, and a testing example, the present invention is further explained. (In the text, part means part by weight.)

Application Example 1

Binapacryl	33.0 parts
Calcium lignin sulfonate	6.0 parts
Alizarine	0.3 part
Basic aluminum acetate	0.3 part
Sodium hydroxide (for adjusting pH)	0.3 part
Ethylene glycol	6.0 parts
Hydroxyethyl cellulose	0.5 part
Water	53.6 parts

Binapacryl, calcium lignin sulfonate, alizarine, basic aluminum acetate, sodium hydroxide, ethylene glycol, and 40 parts water were mixed as a suspension, put into a crushing tank of attriter (made by Mitsui-Miike Works, MA-1S type), and crushed for 2 h by applying stainless steel balls with a diameter of 4 mm as crushing media. A solution in which hydroxyethyl cellulose was dissolved in the remaining water was added to the suspension obtained and remixed, so that an aqueous suspension of binapacryl was obtained. Its pH was 7.5.

Referential Example 1

Similarly to Application Example 1 except for excluding 0.3 part alizarine and 0.3 part basic aluminum acetate and adding the same weight of water in the prescription of Application Example 1, an aqueous suspension of binapacryl was obtained. Its pH was 7.6.

Referential Example 2

Similarly to Application Example 1 except for excluding 0.3 part sodium hydroxide and adding the same weight of water in the prescription of Application Example 1, an aqueous suspension of binapacryl was obtained. Its pH was 3.7.

Referential Example 3

Similarly to Application Example 1 except for reducing the sodium hydroxide to 0.2 part and adding the same weight of water in the prescription of Application Example 1, an aqueous suspension of binapacryl was obtained. Its pH was 5.0.

Referential Example 4

Similarly to Application Example 1 except for excluding the hydroxyethyl cellulose and adding the same weight of water in the prescription of Application Example 1, an aqueous suspension of binapacryl was obtained. Its pH was 7.5. /4

Testing Example 1

The aqueous suspension of Application Example 1 of the present invention and the aqueous suspensions obtained in Referential Examples 1-4 were added up to a height of 7 cm to a cylinder with an inner diameter of 4.5 cm and a height of 8 cm, and a polyethylene plug was put on it and held for 2 weeks in an isothermal chamber at 50°C. The numerical value obtained by dividing the height (cm) of the soft coagulated particle layer in the aqueous suspension by 7 was a sedimentation volume ratio, and the cylinder was manually shaken and mixed. When the soft coagulated particle layer exhibited the flowability again, the property was assumed as a flow re-dispersibility. Also, the upper layer of the above-mentioned coagulated particle layer was a transparent aqueous dispersed layer. The evaluation of the flow re-dispersibility was expressed by the following references.

[o]: The aqueous suspension immediately exhibits the flowability simply by lightly shaking the cylinder.

[Δ]: The flowability is not shown unless the cylinder is strongly shaken and mixed.

[x]: A strong hard caking is formed at the bottom of the cylinder, and the flowability is not shown, even by strongly shaking and mixing the cylinder.

Table I

	Sedimentation volume ratio		Flow re-flowability	
	Initial stage with a lapse of time	Heating test (50°C, for 14 days)	Initial stage with a lapse of time	Heating test (50°C, for 14 days)
Application	1.00	0.92	o	o
Example 1				
Referential	1.00	0.55	o	X
Example 1				
Referential	1.00	0.56	o	X
Example 2				
Referential	1.00	0.75	o	Δ
Example 3				
Referential	1.00	0.70	o	Δ
Example 4				

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